PATENT SPECIFICATION

815,264

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COMPLETE SPECIFICATION

The Production of White Oil from used Oils

We, GESELLSCHAFT FUR FORSCHUNG UND PATENTVERWERTUNG, a Bordy Corporate organized under the laws of Switzerland, of 111 Freiestrasse, Basel, Switzerland, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the production of white oils and other purified oils e.g., insulating oils, from used lubricating oils.

The production of white oils is to-day substantially based on the same process that has been applied for decades, namely by close refining of specially selected petroleum distillates by means of acids, subsequent bleaching and separation of solid paraffins. While the various stages, of which only the most essential have here been listed, have been developed to a high degree of perfection, the entire process is costly and complicated. The process is similar in principle and in starting materials to that employed in the production of lubricating oils but since in the latter case less refinement is necessary, lubricating oils are obtained in greater yields and at lower cost.

It is an object of this invention to provide a process which enables white oils (and other valuable oils) to be produced from lubricating oil fractions by means of purely adsorptive methods and which, surprisingly is not detrimentally affected by highly impure initial materials, i.e. used lubricating oils (reclaimed oils) can be employed as the starting material.

Processes for the regeneration of reclaimed oil by means of solid adsorbents are known as such (cf. Specifications Nos. 609,688 and 711,487 combining adsorptive treatment and acid refining), but these known processes are designed to restore the original quality of a used oil, i.e. impurities introduced during the use of oils, or formed by chemical transformation, are removed more or less completely.

In Specification No. 609,688, just referred to, the process claimed is a method of regenerating spent internal combustion engine lubricating oils wherein the oils are percolated through bauxite at temperatures above 200°C but below those at which any considerable cracking occurs. It is to be noted that bauxite is the name given to natural hydrated alumina; it contains about 50 to 60% Al_2O_3 and substantial quantities of silica, iron oxide and other materials. It is to be understood that where in the present Specification reference is hereinafter made to alumina, the term is intended to mean substantially pure alumina.

The present invention rests on the discovery that it is possible, by the use of alumina as adsorption agent, to convert used oils, particularly used lubricating oils from internal combustion engines directly into white oils, and similar highly refined oils by the process of chromatographic refining. It is also possible by the same process, and at about the same cost as that with which an improved oil can be obtained from a fairly pure oil, to produce a white oil directly from a used oil by the process of the invention. By this process not only are the impurities removed from spent oil, but further substances which were present in the unused oil are removed.

A lubricating oil broadly comprises

1. "white oil," i.e. pure, largely paraffinic and naphthenic hydrocarbons, and

2. coloured, fluorescent and sulphurous substances which may have perfectly desirable properties for the use as lubricants but which must not be present in a white oil.

It is an object of the present invention to separate these two groups from one another, the said impurities remaining in the second group. Hence the process accordingly enables a product to be produced of which the quality is improved over that of the original oil.

According to the present invention, used lubricating oil (including engine oil) is subjected to chromatographic separation to separate white oil therefrom, the adsorbent being activated alumina in compact layer form. The undesired constituents are adsorbed by the adsorbent and may be burnt during its regeneration. The pure product (white oil) is

[Price 3s. 6d.]

obtained as a direct filtrate in the said chromatographic separation. To obtain oil having a particularly high degree of purity it is desirable to operate with a comparatively low yield of the final product (white oil) relatively to the adsorbent, since in this way the impurities typical of reclaimed oil are substantially all adsorbed together with the coloured, fluorescent and sulphurous substances.

In order to make the process economical, however, several circulation systems in which the adsorbent is moved against the oil flow are arranged in such a manner that the portions of oil remaining in the adsorbent in the one system will be further separated in the next following system after selective elution. The adsorbent remains in its system, is first employed for fine separation and then for preliminary separation, and returns to fine separation after regeneration. In the first stages of the process adsorptive treatment is preformed at an elevated temperature without application of solvent. In the final stages, adsorption is advantageously effected in the presence of a volatile solvent, but this solvent may be dispensed with when the reclaimed oil is of low viscosity. Solvents recommended are the low-boiling paraffin hydrocarbons containing up to 8 carbon atoms. Hydrocarbons of low boiling range are particularly suitable because distillation from the oil can then be performed with ease and without large energy requirements.

The following table shows that by the process according to the invention it is possible to convert a used motor oil into a highly valuable oil which has the oil properties of a product obtained by means of the same treatment applied to a crude oil distillate or a used oil distillate, which are therefore prepared from much purer starting materials.

Oils	Colour "N.P.A." according to A.S.T.M. D—155	Sulphur content (%)	Ash content (%)	Neutralization number (mg KOH/g)	Saponification number (mg KOH/g)	Demulsification number
Used internal combustion engine lubricating oil	black	1.0	0.14	0.3	1.2	1200+
„ —raffinate 0—0.3	1—	0.06	<0.01	0.05	0.2	100
„ „ 0.3—3.0	2½	0.32	<0.01	0.05	0.1	180
Crude oil distillate	4+	1.66	<0.01	0.05	0.1	300
„ —raffinate 0—0.3	1—	0.8	<0.01	0.05	0.1	90
„ „ 0.3—2.0	2½	1.42	<0.01	0.05	0.1	150
Used internal combustion engine lubricating oil distillate	2½		<0.01		0.4	105
„ —raffinate 0.5—2.0	2—		<0.01		0.2	57

(Demulsification number: 1200+, 100, 180, 300, 90, 150 — according to I.P.—19; 105, 57 — according to A.S.T.M. D—157)

The figures t[illegible] the oil designations express the proportionate yield of the oil obtained from the quantity of adsorption agent used.

One method of arranging the individual adsorption stages for the performance of the process according to this invention is indicated in the flow diagram very chemically shown in Fig. 2 of the accompanying drawings. For a better understanding, it is however necessary first to discuss the design of such an adsorption stage. The principle of an adsorption unit is shown in Fig. 1 of the accompanying drawings. Referring to Fig. 1, the oil (O) to be separated enters approximately the middle of the adsorptive unit proper (1). There it travels counter-currently to the solid adsorbent (A) which is fed into the adsorption unit from below. The non-adsorbed portions of the oil leave the lower portion of the unit and are designated as "filtrate" (F). The adsorbent charged with the adsorbable components of the oil is supplied to an extraction unit (2) from the top end of the assembly.

In the extraction unit extraction is performed by means of a highly volatile solvent, preferably a low-boiling paraffin, again according to the counter-current principle. Accordingly, the extraction unit yields the eluted adsorbent (A^1) to which the firmly adsorbed components adhere, and the elution product dissolved in the solvent. It is extracted from the solvent in the distilling column (3) and emerges in a pure form (E). The solvent is returned to the extraction unit. The structural members employed, adsorption and extraction units, both working on the counter-current principle, are among the conventional devices used in the present-day art.

The flow diagram shown in Fig. 2 is a typical example of the process according to this invention. The individual adsorption units (V1—V10) are grouped into four systems (termed "groups" in the following), each group comprising one unit less than the previous one. From the storage tank (T), the oil enters the first adsorption unit of the first group (V1). The filtrate from this unit is then supplied to the second unit and so forth until the pure filtrate emerges from V4. The groups of adsorption units must be so adapted that the final filtrates represent the desired product. This can be achieved by keeping the percentage yield low enough, i.e. by adjusting the oil/adsorptive ratio accordingly. The dimensions of the first group will depend on the quality of the initial oil, the efficiency of the adsorbent and the quality of the final product desired.

The portions of the oil retained by the adsorbent in the first adsorption group are eluted in the various stages, and the elution products supplied to the units in the second adsorption group in such a manner that the product coming from the second stage of the first group is supplied to the fi[illegible]ge of the group and so forth. The following adsorption groups are connected in the same manner. The elution product from stages V5, V8 and V10, which are not, according to the said diagram, supplied to stages of the following group, return to the adsorption units of the preceding group, the oil supplied corresponding to the quality of the relative elution product. The product from V1 returns to the storage tank. The adsorbent moves upward by stages in the various groups, i.e. it moves against the oil. It enters the lowermost stage of a group (which treats the purest product) as a fresh (regenerated) adsorbent, and then moves on by one stage after elution is performed. From the top stage it is fed to a regenerating unit which is a rotary cylindrical kiln in the simplest case. Regeneration is effected by burning in an air stream, the adsorbed impurities supplying the energy required. To preserve the capacity of the adsorbent care should be taken that the temperature does not exceed about 800°C. After regeneration, the adsorbent is returned, after replacement of losses, to the lowest stage of the group.

The arrangement of a total of ten adsorption stages in four adsorption groups according to Fig. 2 is purely by way of example. Both the number of groups and that of the stages within the groups may be varied according to the particular requirements. Moreover, the capacity of the various adsoprtion stages relatively to one another may be varied.

If desired, the adsorption stages may be adjusted so that products of different quality (or for different uses) are obtained from the individual adsorption stages.

It is immaterial for this invention whether or not adsorption in the individual stages of the process is effected in the presence of a solvent. However, it is advantageous to operate, at least in the first adsorption group, without addition of a solvent and to increase the temperature sufficiently for the viscosity of the oil to be lowered so as to permit rapid movement of the adsorbent. In the subsequent adsorption groups, however, a solvent may be employed. In that case, complete removal of the hydrocarbon mixture utilized in extraction will be dispensed with and only the final product separated from the solvent.

WHAT WE CLAIM IS:—

1. A process for the production of white oils which comprises subjecting used lubricating oil to chromatographic separation using activated alumina in compact layer form as the adsorbent material to separate white oil therefrom.

2. A process according to claim 1 wherein the lubricating oil is passed through a plurality of adsorption-separation units.

3. A continuous process according to claim 2 wherein the separating process is performed in several stages in which the oil moves in

counter-current to the adsorbent, the said stages being so combined into groups that each group yields a portion of the oil as a pure product while the portions of oil which have not yet acquired the desired degree of purity are supplied to the next following group and there undergo further treatment according to the same principle.

4. A process according to claim 3 where continuity and a good final product are ensured by a reflux within the system of adsorption units, in which the returning oil is conducted from one group to the immediately preceding one and to those adsorption stages which treat an intermediate product of grade corresponding to the quality of the returning oil.

5. A process according to any of claims 2—4 wherein one or several adsorption units, in particular those operated at the beginning of the process, operate at elevated temperatures.

6. A process according to claim 1 carried out substantially as hereinbefore described with reference to the accompanying drawings.

7. Apparatus substantially as illustrated and described with reference to the accompanying drawings when used for carrying out the process of any of claims 1—5.

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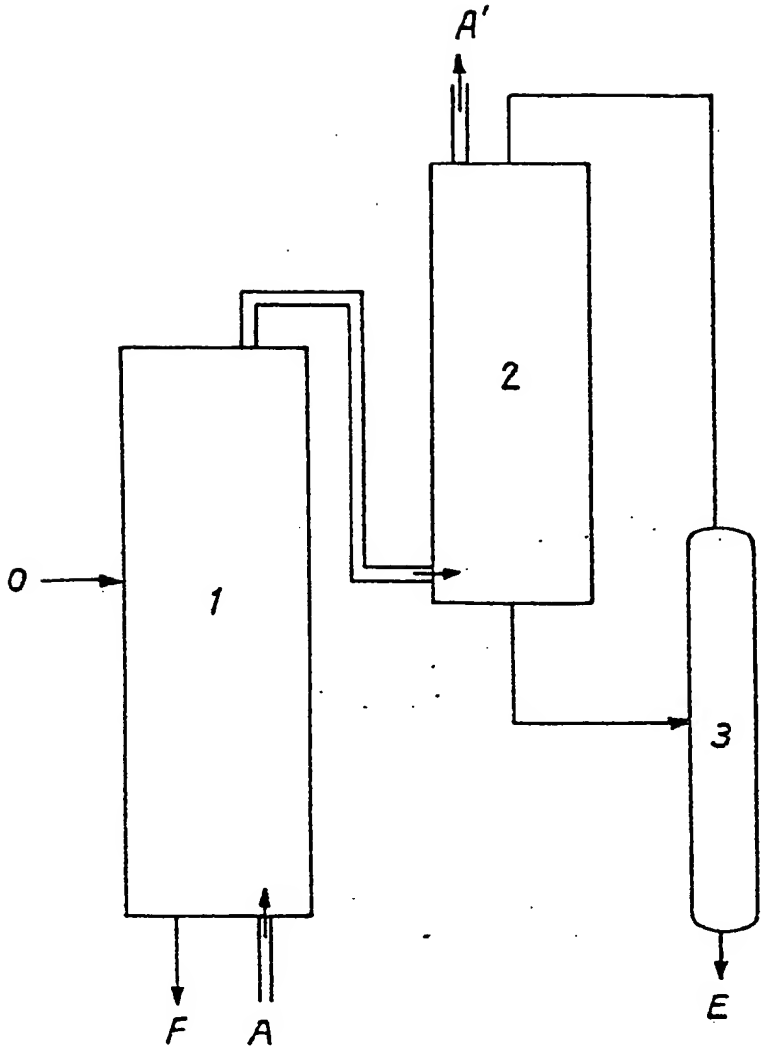

Fig. 1

815,264 COMPLETE SPECIFICATION
2 SHEETS

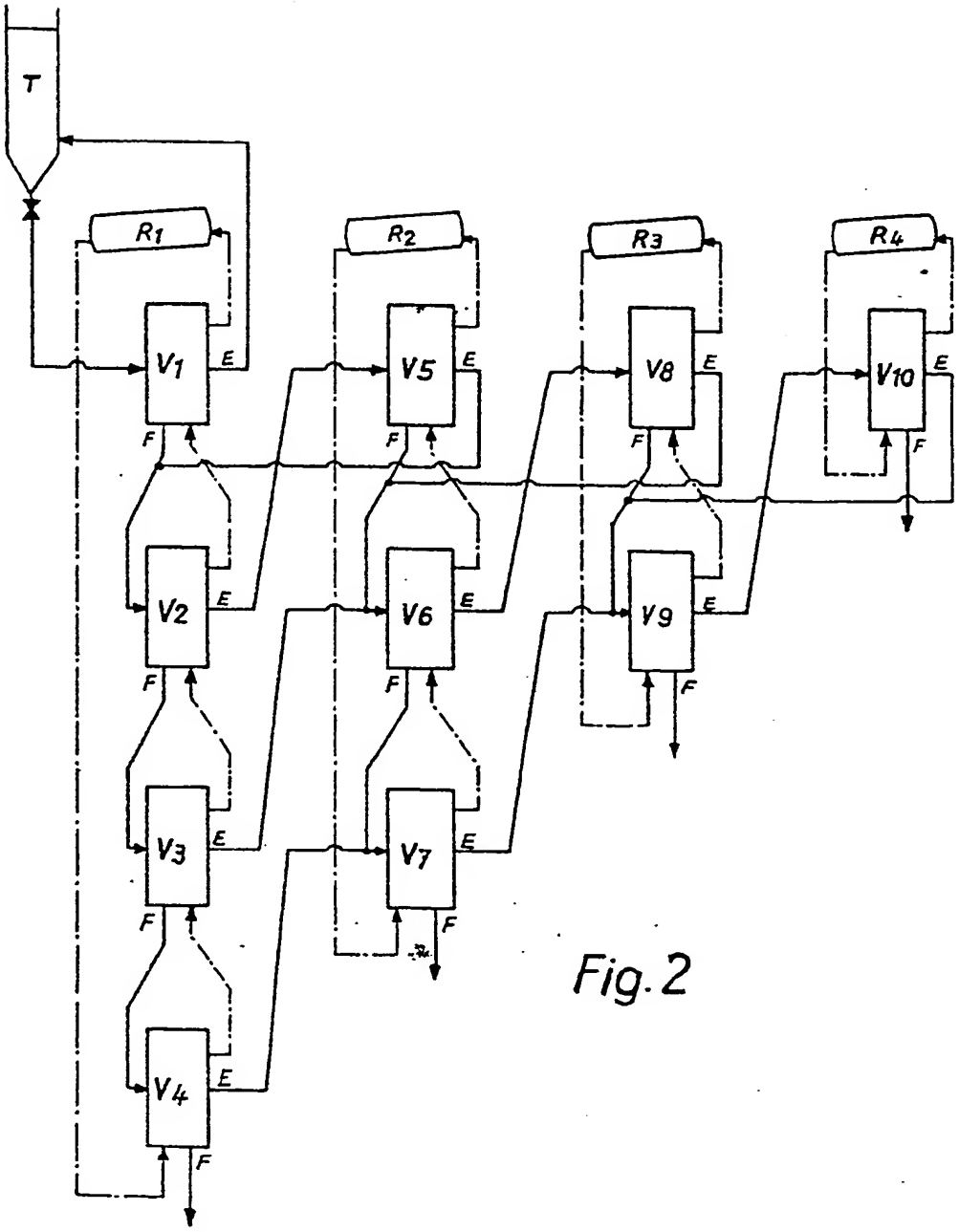

Fig. 2

815,264 COMPLETE SPECIFICATION
2 SHEETS This drawing is a reproduction of the Original on a reduced scale.
SHEETS 1 & 2

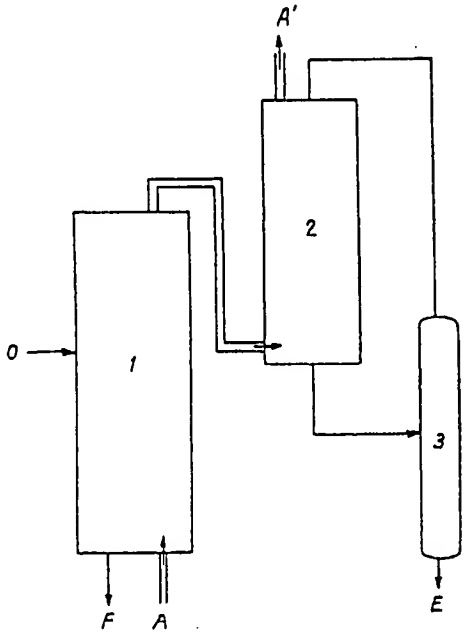

Fig. 1

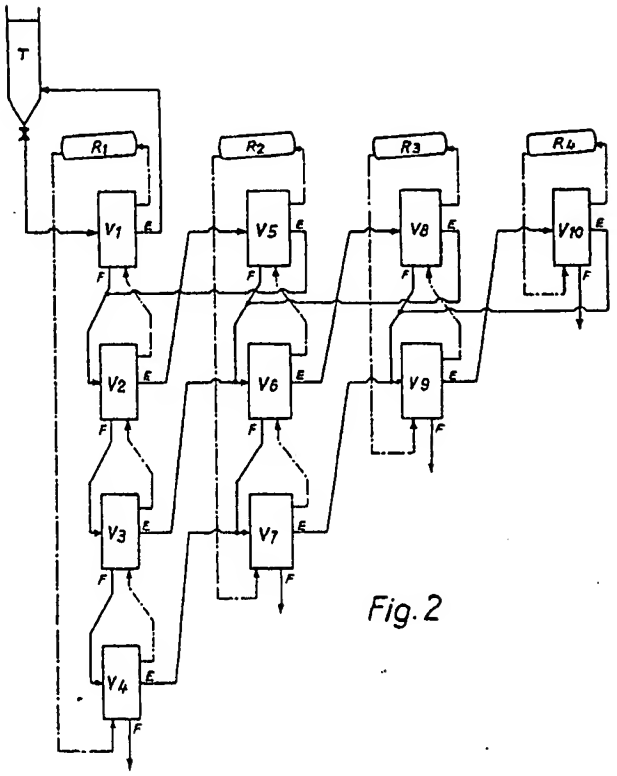

Fig. 2